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Short Communication

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Laves-phase in the China Low Activation Martensitic steel after long-term creep exposure

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Abstract

Creep test at 600 °C under 130 MPa for the China Low Activation

Martensitic (CLAM) steel was performed up to 7913 h in this study. According to the stress level, the crept specimen was divided into three regions in order to investigate the influence of stress on Laves-phase formation. In addition to the expected $M_{23}C_6$ carbide and MX carbonitride, the amount and the size of Laves phase in these three regions on the crept specimen were characterized by transmission electron microscopy. Laves phase could be found in all the regions and the creep stress could promote the formation of Laves phase.

Keywords: China Low Activation Martensitic steel; Creep-rupture; Laves phase; Creep stress

1. Introduction

During the past 40 years, reduced activation ferritic/martensitic (RAFM) steels such as F82H [1], Eurofer97 [2], 9Cr2WVTa and its variants [3, 4, 5] and the China Low Activation Martensitic (CLAM) [6] have been developed in Japan, Europe, US and China, respectively. They are promising candidate materials for first wall and blanket structural application in the future fusion reactors [7]. The precipitates were identified as an important cause of the microstructure instabilities associated with the long-term creep of these high Cr steels [8, 9]. In addition to $M_{23}C_6$-type (M = Fe, Cr, W) carbide and MX-type (M = V, Ta; X = C, N) carbonitride in the tempered RAFM steel [2, 10], Fe_2W Laves phase precipitates are also significant to creep. Conventionally, Laves phase is often considered to influence the microstructure and mechanical properties of those steels [11, 12]. The precipitation of Laves phase with small size should effectively decrease the minimum creep rate, which could compensate the loss of solid solution strengthening at the early stage. However, the later growth and coarsening of Fe_2W would accelerate the creep rate after reaching the minimum creep rate, failing to compensate that loss [13, 14]. However, up to now, precipitation behavior of Laves phase during long term creep exposure has rarely been reported in literature.

The purpose of the present work is to investigate the precipitation behavior of Laves phase in the CLAM steel during long term creep at elevated temperature. The emphasis is on the nucleation, growth and morphology of the Laves phase influenced by the creep stress.

2. Experimental details

The chemical composition of the CLAM steel in the present study was, in wt. %, 0.091% C, 8.93% Cr, 0.49% Mn, 1.51% W, 0.15% V, 0.15% Ta, and Fe in balance. The CLAM steel plate was heat treated by normalizing at 980 °C for 30 min and tempering at 760 °C for 90 min. Then, specimens with 5 mm gauge diameter and 67 mm gauge length were machined from the tempered plate with the axis parallel to the rolling direction.

Creep-rupture test was conducted in air at 600 °C under 130 MPa. The crept specimen after creep fracture was divided into three regions, the necking down region (region A), the uniform plastic deformation region (region B) and the clamp region (region C). Region A was near the creep fracture surface with large deformation, region B had uniform plastic deformation and region C was the part of clamp segment of the crept specimen under low stress during the creep rupture test. Therefore, region C was considered as the aging region. Figure 1 shows the three different regions of a crept specimen.

The microstructures of the different regions on the crept specimen were characterized by using a transmission electron microscope (TEM). The thin foils for the TEM observation were cut from the creep specimens along the transverse cross-section, and then electrochemically polished in a solution of 10% perchloric acid and 90% acetic acid at 11-15 °C and 28-30 V.

3. Results

Figure 2 shows the microstructure of the presently studied CLAM steel before the creep test. The microstructure of CLAM steel was tempered martensite after the heat treatment. $M_{23}C_6$ carbide and MX carbonitride were the main precipitates present in the microstructure. The

$M_{23}C_6$ carbide was precipitated at the prior austenite grain boundaries, as well as along the subgrains and lath boundaries. The primary morphology of $M_{23}C_6$ precipitates mainly includes rod-shaped and spherical, and the average size is about 80 nm. MX carbonitride was formed within the subgrains with size of about 20-40 nm. Laves phase was not detected after the heat treatment.

The crept specimen at 600 °C under 130 MPa for the duration of 7913 h was chosen for study. With varying distance from crept fracture, the lath martensite in CLAM steel showed different degrees of recovery and recrystallization.

The size of $M_{23}C_6$ precipitates in the crept specimen was larger than that in the tempered steel. The presence of Laves phase in the crept specimen was investigated by TEM. Using the results of the microstructural study, the regions had the following features:

(i) For region C (Figure 3c), this part of the crept specimen was little affected by the creep stress. The morphology of martensite in this part was observed by TEM. The width of martensite lath in region C was about 0.62 μm. The Laves precipitates in this region could reach large dimensions, with average diameter of about 0.32 μm.

(ii) For region B (Figure 3b), the width of the martensite lath and fragmentation of subgrain were observed. Meanwhile, the Laves phase was smaller than that in region C, but the number was larger than that in region C.

(iii) For region A (Figure 3a), the CLAM steel was characterized by its fine subgrain structure with a high density of free dislocations within the subgrains. Compared to regions B and C, the average size of Laves phase was the smallest, but the amount of Laves phase was the largest.

Meanwhile, precipitates of MX were found to be highly resistant to coarsening during the creep.

4. Discussion

In this work, the crept specimen of CLAM steel was divided into three regions, and Laves phase precipitation in each region was studied by microstructural observation.

In the tempered CLAM steel, there were only two types of precipitates in the matrix, $M_{23}C_6$ carbide and MX carbonitride. The Laves phase, which would occur during the long-term creep exposure, did not appear. In our previous work [10, 15], the precipitation of Laves phase appeared in the steel matrix after aging at 600 °C for 1100 h. In the present study, the specimen of CLAM steel was crept at 600 °C under 130 MPa for duration of 7913 h, and the precipitation of Laves phase could be observed in all the three regions on the crept specimen. It has been found that Laves phase nucleated mostly at martensitic lath boundaries [16], subgrain boundaries and prior austenitic grain boundaries [17]. The number and size of Laves phase were different in three regions on the crept specimen.

Region C was the part of the crept specimen under low stress, which was regarded as an aged region. The width of martensite lath increased compared with that in the tempered steel, but the feature of martensite lath was kept. The addition of W in the steel could slow down the growth of lath width and the annihilation of dislocations in lath interior [16].

The new phase (Laves phase) would nucleate at grain boundaries by consuming the W element around it and hence decreased the concentration of W, resulting in a reduction of the phase transformation free energy, which would raise the critical nucleation energy and reduce the nucleation rate. Though the tempered martensitic lath structure would be stable at the elevated temperatures without stress, its recovery took place substantially during the creep [16]. The dislocation density decreased and the subgrains grew during the creep of 9-12 % Cr steels under suitable ranges of stress and temperature [18]. The creep stress could accelerate the microstructural evolution, including the recovery of excess dislocations and the growth of martensite lath subgrains.

The dragging effect on dislocations by W atoms under stress was considered to be one of important factors for the enhancement of precipitation and growth of the Laves phase [19]. The dragged dislocations could provide the nucleation sites for the Laves phase. The growth rates of Laves phase in regions A and B were much lower than that in region C. It was ascribed to the fast nucleation of Laves phase, which consumed much W in solid solution and resulted in the decrease of W concentration. Then the Laves phase would grow slowly with low W concentration. However, when the rate of W atom diffusion is increased by high temperature, Laves phase in the region affected by stress might grow faster compared to other regions. This effect was confirmed in a recent paper in another steel of the heat-resistant type [20].

5. Conclusions

The crept CLAM steel at 600 °C under a constant load of 130 MPa for 7913 h was investigated. The crept specimen was divided into three regions according to the different stress levels. The creep stress could enhance the microstructure recovery. Laves phase was found in all the regions on the crept specimen, but showed different features along the length of crept specimen. Compared with the region under low stress, the density of Laves phase was high, while its average size was small near the neck down region. The creep stress enhanced the formation of Laves phase during creep due to the acceleration of nucleation rate of Laves phase under creep stress in the early stage.

Acknowledgements

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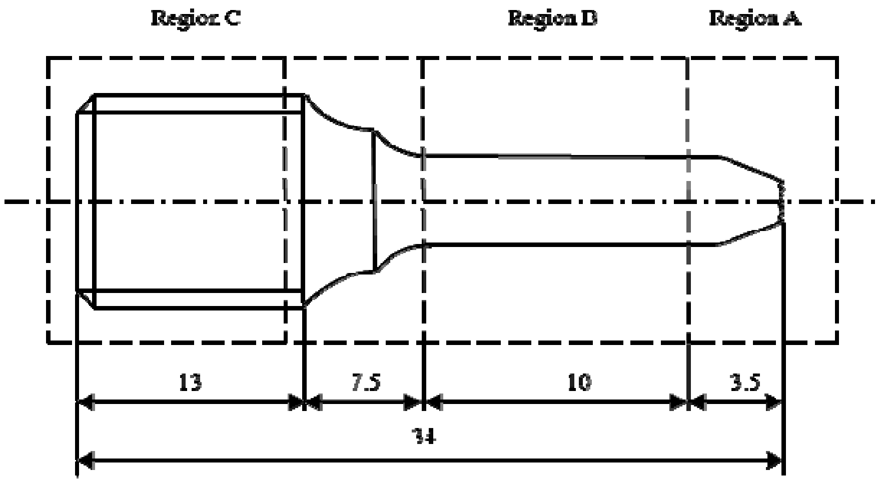
Figure captions

Figure 1. Sketch of different regions in a crept specimen. Dimension in milimetres.

Figure 2. Microstructure of the tempered CLAM steel observed on TEM.

Figure 3. Microstructures of three regions of crept specimen. (a) Region A; (b) region B; (c) region C; (d) electron diffraction patterns of Laves phase.



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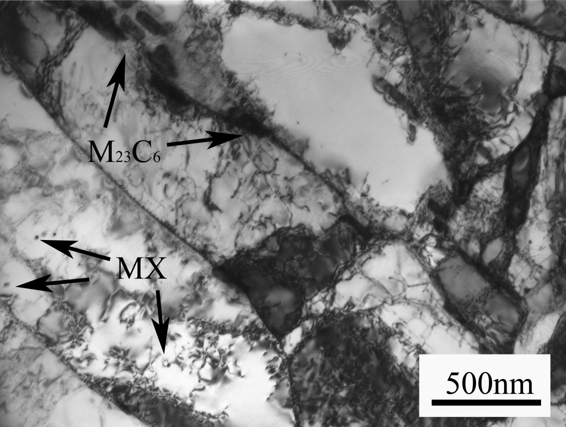
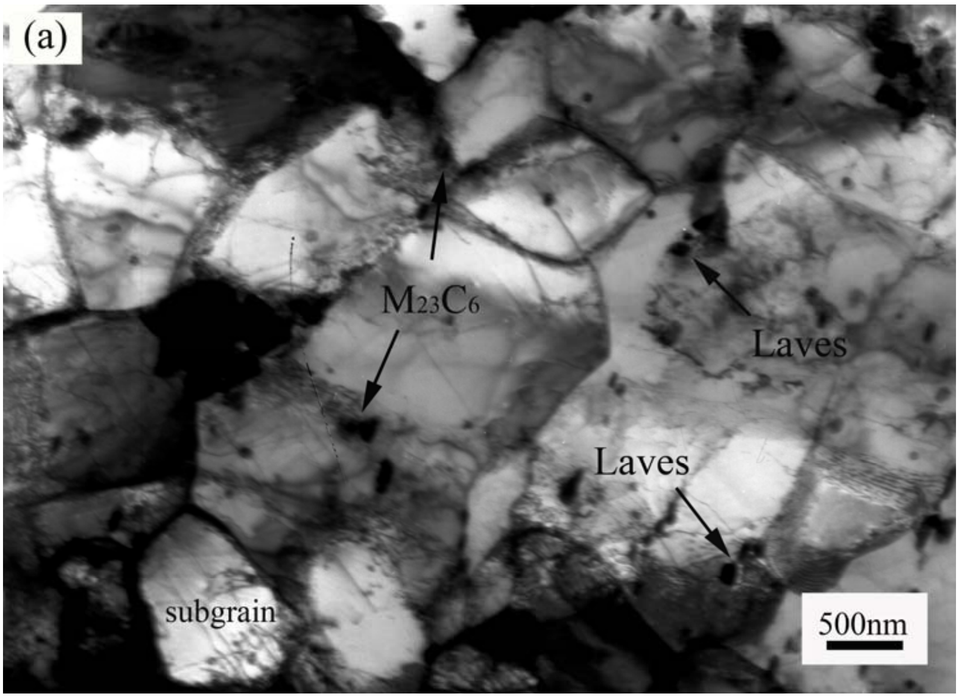
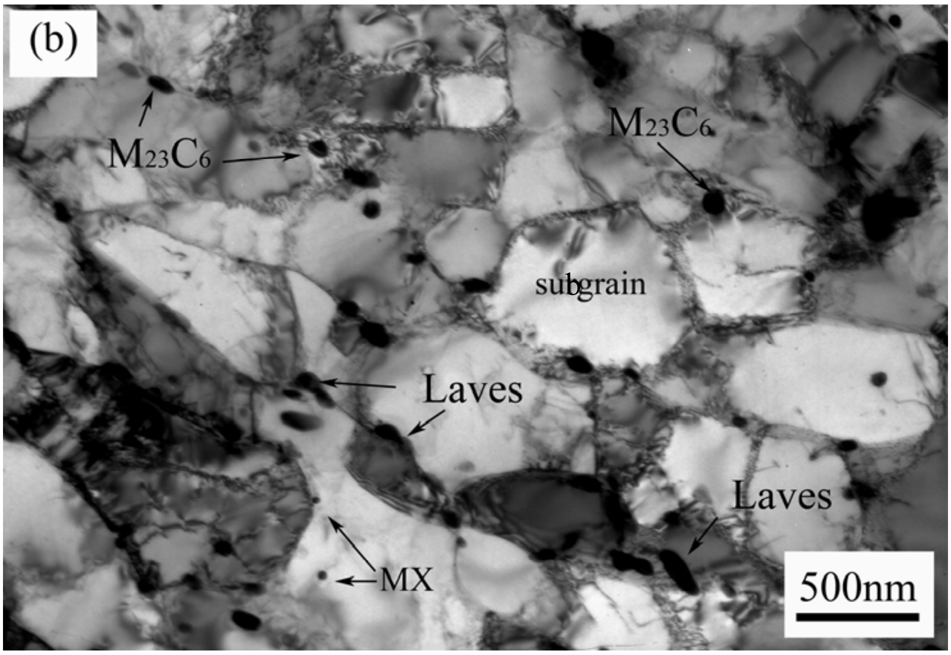
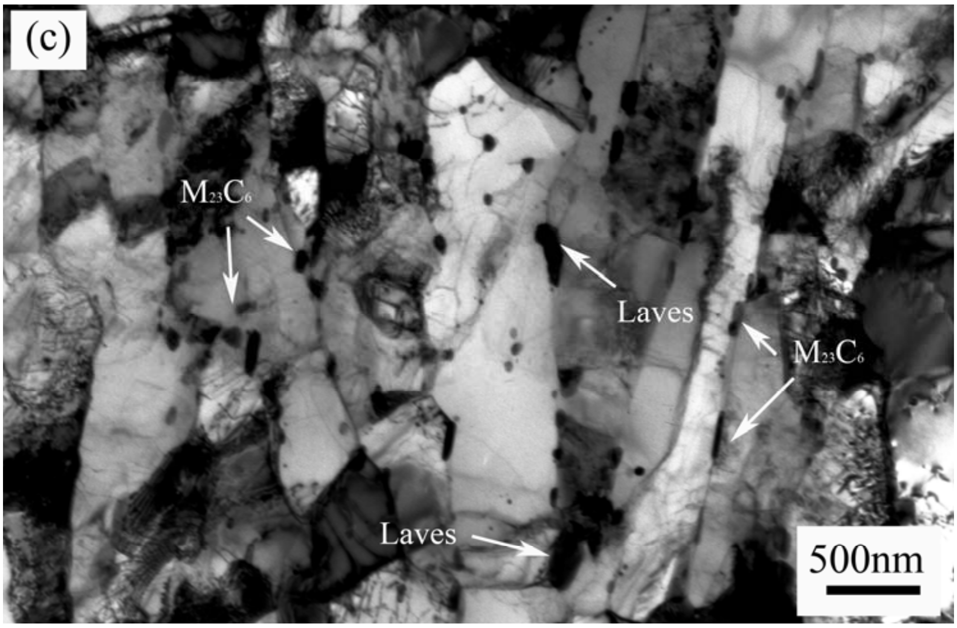

M23C6
MX
500nm

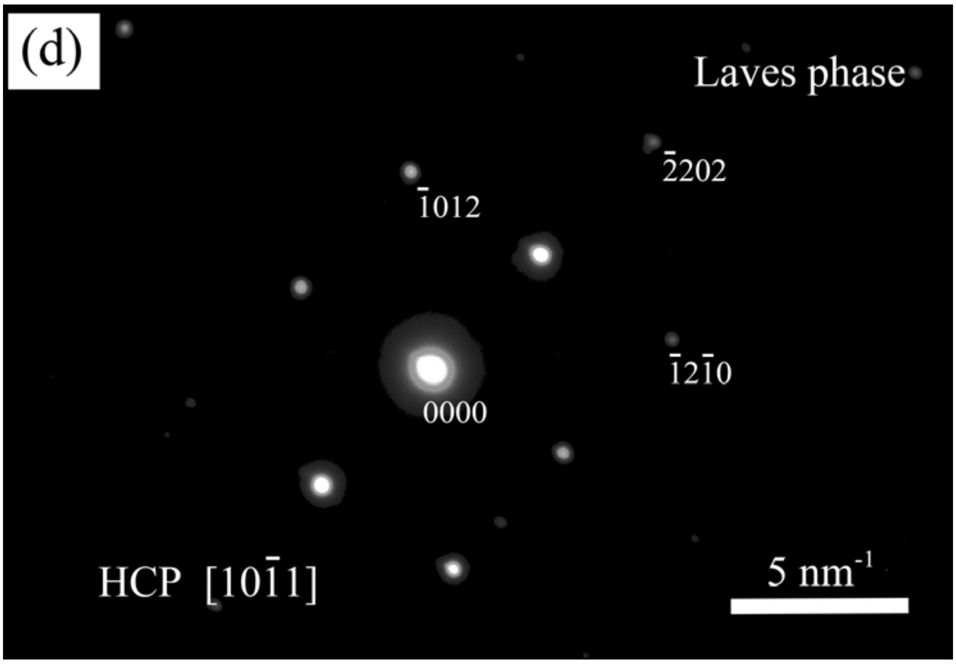


(a)
M23C6
Laves
Laves
subgrain
500nm



(b)
M23C6
M23C6
subgrain
Laves
Laves
MX
500nm





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